

FM001 Manual water quality sampling for total suspended solids and nutrients

Water Quality and Investigations – Environmental Monitoring and Assessment Science

This document has been modified by Terrain NRM for the Wet Tropics Major Integrated Project

1 Purpose and scope

This document provides guidance on the process of manually collecting water samples and submitting them for analysis as part of the Wet Tropics Major Integrated Project (WTMIP).

The method outlined in this document applies whenever water quality samples are manually collected and submitted to a laboratory for analysis of total suspended solids (TSS) and nutrients. Manual sample collection refers to the process of collecting water samples directly into sample bottles without the use of an intermediate sampling container (e.g. automatic pump sampler).

2 Training, competency and responsibilities

Program staff, regional officers and external stakeholders participating in the collection of samples are provided with training in water sampling methods. Records of participant competency are maintained within the Terrain Natural Resource Management (NRM) file management system (SharePoint).

Table 1 Roles and responsibilities of WTMIP program participants

Position	Responsibilities
WTMIP Water Quality Project Officers (Leader/Technician)	Ensure methods described in this field method are followed Provide resources for continued development of this method
WTMIP program staff (including regional officers and external stakeholders)	Follow the methods described in this document Continually review and develop the methods where appropriate

3 Workplace health and safety

Field based work activities must adhere to Terrain NRM WHS requirements. The following procedures and equipment must be considered prior to undertaking fieldwork:

- Terrain NRM Water Quality Monitoring Risk Assessment
- Trip plan, including call-in schedule and emergency response procedures
- Communication devices required for the trip (e.g. mobile phone, SPOT device, EPIRB)
- First aid kit
- Personal Protective Equipment, such as safety boots, pants, long sleeved shirt, hat, high visibility clothing.

4 Equipment

A list of equipment required for the manual collection of water samples is provided in Appendix A. The quantity of items required will be determined by the number of samples that will be collected during the field trip.

Sample bottles, syringe and filters are shown in Figure 1 and Figure 2. A summary of the bottle types, water quality analyses, minimum sample volume, and sample processing and storage conditions are provided in Table 2.



Figure 1 Sample bottles for dissolved nutrients, total nutrients and total suspended solids (left-right)



Figure 2 Syringe, filter and pre-filter

Table 2 Bottle type¹, volume, processing and storage summary

	Bottle A	Bottle D	Bottle E
Analysis type	- Total Suspended Solids -	- Total Nutrients (Total Nitrogen & Phosphorus) -	- Dissolved Nutrients (Phosphate phosphorus as P, total dissolved phosphorus as P, dissolved inorganic phosphorus as P, ammonium nitrogen as N, total dissolved nitrogen as N, oxidised nitrogen as N, nitrate as N, nitrite as N, dissolved Keldahl nitrogen as N.
Bottle volume	500mL	100mL	50mL
Minimum sample volume	250mL	40mL	40mL
Filter sample?	No	No	Yes
Headspace required	No	Yes	Yes
Sample storage	Chilled (1-4°C)	Refrigerate immediately & freeze (-20°C) within 24 hours	Refrigerate immediately & freeze (-20°C) within 24 hours
Store sample in the dark?	Yes	No	No
Maximum holding	7 days	24 hours chilled or 28 days frozen	24 hours chilled or 28 days frozen

¹ Each bottle must be made of polyethylene

5 Method

To limit the risk of sample contamination, water samples should be collected directly into the total suspended solids bottle and the total nutrients bottle by sequentially immersing each bottle into an area of representative flow where the water is well mixed. It is important that samples are collected upstream of significant in-stream structures and known point sources of pollutants.

The recommended method for water sample collection for the WTMIP is to fill sample bottles using an extendable sampling pole with a bottle clamp attachment. Using an extendable sampling pole will assist in reaching an area of representative flow and reduce the risk to the sampler associated with entering the water (i.e. deep water, strong currents or areas potentially inhabited by crocodiles).

For more information on field collection methods, see the Monitoring and Sampling Manual (DES 2018).

5.1 Preparation for sample collection

To mitigate the risk of sample contamination **it is mandatory that staff wear non-powdered gloves during all stages of sample collection** and avoid touching the mouths of bottles, undersides of lids and ends of syringes and filters.

1. Prepare the work area to ensure it is free of contaminants and contents of the sampling kit are easily accessible. Remove sample bottles and syringe from the sampling kit. Filters should be placed on the work area though not opened until required.
2. Visually inspect all items ensuring the lids of all sample bottles are firmly secured and packaging of syringes and filters are sealed. Discard all items if the integrity of the packaging is compromised or potential for contamination has occurred.
3. Find the package of pre-labelled sample bottles that corresponds with the name of the site at which samples are about to be collected. The second field staff member is to check that the correct bottle pack has been selected.
4. Complete all mandatory documentation (e.g. electronic Chain of Custody form). Mandatory information includes: site name, date, time, sampler's name and the bottles collected.

5.2 Collecting water samples

5.2.1 Collecting total suspended solids and total nutrient samples using an extendable sampling pole

1. Identify a representative section of the stream to collect the water sample/s.
2. Extend the sampling pole to the required length, ensure it is clean, and with clean gloves on, place the total suspended solids bottle into the clamp attachment.
3. Remove the lid of the sample bottle, taking care not to touch the inside surfaces of the lid or the sample bottle.
4. Position the sample bottle above the water where the sample will be collected. With the neck of the bottle facing down, submerge it in to the main flow of the stream (avoiding surface contamination) to a depth of approximately 0.3 m.
5. Rotate the submerged sample bottle upstream into the direction of flow and allow the bottle to fill approximately 1/3 with stream water.
6. Remove the bottle from the water and replace the lid on the sample bottle.

7. Shake the sample bottle to rinse, ensuring the inside of the bottle and lid come into contact with the stream water. Discard this water downstream of the sampling area.
8. Repeat steps 3 to 7 so the sample bottle is rinsed twice with stream water.
9. To fill the sample bottle, repeat steps 3 to 5, this time allowing the sample bottle to fill completely.
10. Replace the lid, ensuring a good seal.
11. Repeat steps 3 to 10 to fill the total nutrients bottle, leaving 10% free space to allow for the water to expand on freezing and screw the lid on tightly.
12. Proceed to Section 5.3 to filter into the dissolved nutrients bottle if a water sample is required for analysis of dissolved nutrients. If this is not required, ensure all information on the sample bottles and mandatory documentation has been completed and is correct, and place the sample bottles in an esky with sufficient ice bricks, or a car fridge, to rapidly chill the sample to below 4°C.



Figure 3 Sampling pole with the total suspended solids bottle inverted in preparation to collect a water sample

5.2.2 Sample collection by hand

If an extendable sampling pole cannot be used, the sample can be collected by hand if safe to do so. When collecting samples by hand it is important to orientate the sample bottle upstream into the current to avoid contamination from the sampler, their hands or material that may resuspend due to disturbance of the substrate.

5.3 Filtering into the dissolved nutrients bottle

The process to fill the dissolved nutrients bottle requires that water be decanted from the total nutrients bottle into a syringe which is subsequently filtered into the dissolved nutrients bottle. In undertaking this process, both the total nutrients bottle and the syringe are considered intermediate containers and controls must be in place to mitigate the additional risks of sample contamination.

The following procedure should be followed to process all water samples for analysis of filtered nutrients.

1. Attach a filter (and pre-filter if necessary). Connect filter without touching end of the filter by using the plastic packaging.

2. Remove plunger from the syringe – avoid touching the internal surfaces of the barrel and plunger.
3. Shake the total nutrients bottle thoroughly to resuspend solids and fill the syringe with at least 40 mL of sample water and replace the plunger (Figure 4).
4. Discard the first 2 mL of sample water pushed through the filters as a filter rinse.
5. Remove the lid from the dissolved nutrients bottle and filter approximately 5 mL of sample into the bottle.
6. Replace the lid and shake vigorously ensuring all surfaces of the dissolved nutrients bottle and lid come into contact with the water then discard waste water downstream.
7. Repeat steps 5 and 6 to rinse the dissolved nutrients bottle **twice**.
8. Filter the remaining sample into the 'E' bottle (Figure 4). If filtering becomes difficult, change the filter (and pre-filter), making sure each set of filters are rinsed with 2 mL of sample water before continuing to filter the sample.
9. Replace the lid, ensuring a good seal and freeze the dissolved nutrients bottle immediately.

During filtering it is important that staff be patient as the process may become difficult if samples contain a high concentration of suspended solids. Change the filters as frequently as necessary to ensure the minimum required filtered sample volume is obtained.



Figure 4 Example of pouring from the D bottle into the syringe (left); and filtering the sample into a DES dissolved nutrients bottle using a pre-filter (right)

5.4 Sample preservation

When in the field all samples should be immediately placed in an esky containing sufficient ice bricks to rapidly cool all samples to below 4°C and the esky put in a cool dark place. Portable refrigeration/freezer units may be used where available, however samples for analysis of total suspended solids must not be frozen at any stage.

On returning to the office all total suspended solids bottles must remain chilled below 4°C in a designated sample refrigerator. Total nutrients bottles and dissolved nutrients bottles must be frozen (-20°C) and remain frozen during transport to the laboratory. For further information refer to Table 2.

5.5 Sample transport

During field work or when sending samples to the laboratory it is important to ensure an adequate number of ice bricks are placed in eskies with the samples. It is advised that chilled total suspended sediments bottles be transported in a separate esky to frozen total nutrients bottles and dissolved nutrients bottles as this allows

greater control over the preservation conditions – a higher ratio of ice bricks may be placed in eskies containing the frozen samples to prevent them from thawing.

In order to minimise contamination through leakage and sample bottles breaking, eskies should be clean, bottle lids must be on tight, glass bottles (if collecting pesticide samples) should be wrapped in padded sleeves and bottles should be packed upright.

It is critical that samples are transported to the laboratory as soon as practical. Sample transport will be coordinated by the WTMIP Water Quality Project Officers (Leader/Technician) so that samples are received by the laboratory for analysis within holding periods.

6 Water quality control samples

Quality control sampling for the WTMIP follows guidance contained in Monitoring and Sampling Manual (DES 2018). The quality control samples required for this program, and the frequency of their collection, are outlined in Table 3.

The transport blank, field spike and the laboratory-grade ultrapure water ('Milli-Q' water) for the field blank must be prepared by a NATA accredited laboratory. It is the responsibility of samplers to ensure they have an adequate supply of quality control samples.

Authorisation must be received from WTMIP Water Quality Project Officers for any change to the frequency outlined below. Organisations and staff are encouraged to discuss implementation of quality control sampling prior to commencement of each wet season in order to evaluate the risks of sample contamination.

For information regarding the collection of quality control samples, refer to *FM010 Quality assurance and quality control procedures associated with the collection of water samples*. Further information is also available in the Monitoring and Sampling Manual (DES 2018).

Table 3 The rate of quality control sample collection

Quality control sample	Frequency of collection
Field blank	4 per site per year (2 during ambient conditions, 2 during wet conditions)
Transport blank	4 per site per year (2 during ambient conditions, 2 during wet conditions)
Field spike	4 per site per year (2 during ambient conditions, 2 during wet conditions)
Replicates	4 per site per year (2 during ambient conditions, 2 during wet conditions)

7 Sample documentation

All water quality samples must be accompanied by supporting documentation. Laboratory services for the WTMIP water quality program are provided by Cairns Regional Council (CRC) Water Laboratory. The CRC Water Laboratory has an established Laboratory Information Management System (LIMS) that produces electronic Chain of Custody forms already populated with the sample details. It is the responsibility of the sampler to ensure they understand how to record the relevant information using the CRC Water Laboratory sample submission process, and to contact the WTMIP water quality team for advice if required. It is also the responsibility of the sampler to submit the CRC Water Laboratory form to the WTMIP Water Quality Project Officers.

7.1 WQI Sample Submission Sheet

The CRC Water Laboratory Chain of Custody form (Figure 4) is used to manually document sample metadata. Multiple samples can be recorded on the same Chain of Custody form.

Mandatory fields of the Chain of Custody form to be completed by the sampler are:

- Samplers' name and email address
- Date and time sampled
- Use the drop-down options to record whether the sample is part of a routine or event-based sampling run, and the flow conditions (i.e. base-flow or, if high-flow, whether the water level was rising, steady, falling or unknown)
- Physico-chemical measurements taken with the EXO2 (i.e. pH, electrical conductivity, turbidity, nitrate, temperature and dissolved oxygen) for each sample collected.

An example of a CRC Water Laboratory Chain of Custody form is provided below in Figure 5.

General comments, including quality control sample details, photo numbers, any change in sampling process or location, and other notes that may be relevant for the interpretation of results are to be made on the separate WTMIP field proforma sheet.

Cairns Regional Council Laboratory Services



Please fill in the blue boxes below and email to laboratory@cairns.qld.gov.au

Please deliver samples to:
**Cairns Regional Council
 Laboratory Services
 38 MacNamara St
 MANUNDA QLD 4870**

Phone:
 Fax:
 Web:

Customer Details

Company Name:
 Project Number:
 Contact Name:
 Email Address:
 Project Description:

#	Sample Number	Sampling Point	Description	Sampled Date/Time	Routine/Event Based?	Flow Condition	pH	uS/cm	FNU	mg/L	%	deg C	container numbers
								Electrical Conductance	Turbidity	Nitrate	Dissolved Oxygen	Temperature	
1	625453	NONE	MIP LSM J1										625463 625464 625465
2	625454	NONE	MIP LSM J2										625466 625467 625468
3	625455	NONE	MIP LSM J3										625469 625470 625471
4	625456	NONE	MIP LSM J4										625472 625473 625474
5	625457	NONE	MIP LSM J5										625475 625476 625477
6	625458	NONE	MIP LSM J6										625478 625479 625480
7	625459	NONE	MIP LSM J7										625481 625482 625483
8	625460	NONE	MIP LSM J8										625484 625485 625486
9	625461	NONE	MIP LSM J9										625487 625488 625489
10	625462	NONE	MIP LSM J10										625490 625491 625492
11													625447 625448 625449
12													625450 625451 625452

Figure 5 Cairns Regional Council Water Laboratory Chain of Custody form

8 References

AS/NZS 5667.1:1998, Water quality – Sampling: Guidance on the design of sampling programs, sampling techniques and the preservation and handling of samples, Standards Australia.

AS/NZS 5667.6:1998, Water quality – Sampling: Guidance on sampling of rivers and streams, Standards Australia.

DES 2018. Monitoring and Sampling Manual: Environmental Protection (Water) Policy. Brisbane: Department of Environment and Science Government.

DSITI 2017, FM002 Water quality sampling for total suspended solids and nutrients using an Isco Avalanche automatic sampler

DSITI 2017, FM010 Quality assurance and quality control procedures associated with the collection of water samples

Appendix A Equipment list

Equipment	✓
Sampling pack for each suite of samples as supplied by CRC Water Laboratory, for each sampling site, including: <ul style="list-style-type: none"> ▪ 3 x sampling bottles (500mL bottle for suspended solids, 100mL bottle for unfiltered nutrients and 50mL bottle for filtered nutrients) (Figure 1) ▪ 1 x individually packed and sealed TERUMO 60 mL syringe (Figure 2) ▪ 1 x Sartorius Minisart® 0.45 µm hermetically sealed filter individually packed and sealed (Figure 2) 	
1 x box of spare Sartorius Minisart® 0.45 µm hermetically sealed filters individually packed and sealed (Figure 2)	
1 x box of Sartorius Minisart PLUS® glass fibre pre-filter for sampling waters with high suspended particulate load (Figure 2)	
Non-powdered disposable gloves	
Sample documentation (i.e. electronic CRC Water Laboratory Chain of Custody form loaded on field tablet and hard copy back up) (see Section 9)	
Accurate time equipment (i.e. mobile phone)	
Clean esky/eskies with ice bricks or refrigerator	
Extendable sampling pole with bottle clamp attachment	
Quality control solutions (see Section 6): <ul style="list-style-type: none"> ▪ Laboratory supplied field spike sample ▪ Laboratory supplied transport blank sample ▪ Laboratory-grade ultrapure water ('Milli-Q' water) (2L per sample) for collecting field blank 	
Additional sampling packs for the collection of quality control samples	
Phosphate-free detergent (e.g. Decon90) (to clean extendable sampling pole)	

Version Control

Document	Date	Amendment:	Amended by:	Reviewed by:	Approved by:
History					
Version 1.0			Rohan Wallace	Dr Michael Warne Dr Suzanne Vardy	Dr Christine Williams
Version 1.1		5.3.3 - Major revision of sample processing procedure for dissolved nutrients 5.4 – Amendment to the procedure for collecting samples with an extendable pole 6.1 - Amendment to the field blank procedure	Rohan Wallace	Dr Rachael Smith Rae Huggins	Dr Michael Warne
Version 1.2		Update of work group name	Rohan Wallace		
Version 1.3		Update of template and division name	Rohan Wallace		
Version 1.4		Update of template and department name	Rohan Wallace		
Version 1.5		Update of template	Zackary Severino		
Version 2.0	July 2017	Update of structure Review of QA/QC procedures Addition of Obzervr	Leigh Anderson	Rohan Wallace	
Version WTMIP 1.0	October 2018	Modified to include details specific to the Wet Tropics Major Integrated Project	Alicia Buckle Emma-Lee Harper (Terrain NRM)	Dr Ryan Turner Rohan Wallace	

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